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Indian Standard "पुनर्पट्ट १६६५"

SPECIFICATION FOR SOLUBLE STARCH PHOSPHATE (EDIBLE GRADE)

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INDIAN STANDARDS INSTITUTION MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG NEW DELHI 110002

Indian Standard

SPECIFICATION FOR SOLUBLE STARCH PHOSPHATE (EDIBLE GRADE)

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Indian Standard

SPECIFICATION FOR SOLUBLE STARCH PHOSPHATE (EDIBLE GRADE)

0. FOREWORD

- **0.1** This Indian Standard was adopted by the Indian Standards Institution on 23 June 1983, after the draft finalized by the Edible Starches and Starch Products Sectional Committee had been approved by the Agricultural and Food Products Division Council.
- 0.2 Soluble starch phosphate, a modified starch, is manufactured in the country on commercial scale. It is used in the food industry as a thickener, filler, binder, stabilizer and flavour carrier. It is valued in various food products due to its thickening, rheological and moisture retention properties. It gives a clear non-congealing paste, with marked reduction in the tendency of the cold paste to set back on standing and absence of syneresis on long storage.
- 0.2.1 The inherent physico-chemical properties enables its use in different food industries especially bakeries, confectioneries, and in the manufacture of jellies, ice creams, beverage emulsions, flavouring agents and most other foods and edible products and also pharmaceuticals.
- 0.3 In the preparation of this standard due consideration has been given to Prevention of Food Adulteration Act, 1954 and the rules framed thereunder, and the Standards of Weights and Measures (Packaged Commodities) Rules, 1977. However, this standard is subject to the restriction imposed under these wherever applicable.
- 0.4 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS: 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

^{*}Rules for rounding off numerical values (revised).

1. SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for soluble starch phosphate (edible grade).

2. DEFINITION

2.1 Soluble Starch Phosphate — Generic term indicating starch ester in which some or all of the available hydroxyl groups of the starch have been esterified by phosphate groups.

3. RAW MATERIAL

3.1 The product shall be manufactured from edible maize starch, edible tapioca starch or any other suitable edible starch by the use of suitable phosphatizing agents.

4. REQUIREMENTS

- 4.1 Description—The product shall be in the form of fine dry powder, either white or creamy in colour and bland in taste. It shall readily dissolve in water. It shall be free from rancidity, adulterants, insect or fungus infestation and fermented, musty or other objectionable odours. The material shall be free from dirt and other suspended and extraneous matter.
- 4.2 Particle Size When tested by the method prescribed in 3 of IS: 4706 (Part 1)-1978*, all the material shall pass through 150 µm IS sieve [see IS: 460 (Part 1)-1978†].
- **4.3 Hygienic Conditions** The material shall be processed and packed under hygienic conditions (see IS: 2491-1972‡).

^{*}Methods of test for edible starches and starch products: Part I Physical methods (first revision).

[†]Specification for test sieves: Part I Wire cloth test sieves (second revision), ‡Code for hygienic conditions for food processing units (first revision).

4.4 The material shall also comply with the requirements given in Table 1.

TABLE 1 REQUIREMENTS FOR SOLUBLE STARCH PHOSPHATE

SL	CHARACTERISTIC	REQUIREMENT	METHOD OF TEST REF TO		
No.			Appendix of this standard	Appendix of IS: 873-1974*	Cl No. of 1S: 4706 (Part 2)- 1978†
(1)	(2)	(3)	(4)	(5)	(6)
i)	Moisture, percent by mass, Max	10.0	_		4
ii)	Acid insoluble ash, percent by Mass, Max	0.2		_	8
iii)	Water-insoluble matter, percent by mass, Max	1.0	A		
iv)	pH of aqueous extract	6 to 8	_	_	13
v)	Phosphorus content, percent by mass, Max	0.5	В		_
vi)	Viscosity, of a 2 percent (m/m) solution in Pa. s (as measured with Brookfield synchrolectric viscome Min	40×10 ⁻⁸	c		-
vii)	Arsenic mg/kg, Max	3	_	D	
viii)	Heavy metals (as Pb), mg/kg, Max	10	D	•	

Note-1 cP (centipoise) = 10^{-3} Pa.s (Pascal second).

5. PACKING

5.1 Unless otherwise agreed to between the purchaser and the supplier the material shall be packed in clean, sound and new high density polyethylene (HDPE) woven bags or A-twill jute bags (see IS: 1943-1964*) lined with polyethylene. The mouth of each bag shall be either machine stitched or rolled-over and hand stitched.

^{*} Specification for liquid glucose (first revision).

⁺Methods of test for edible starches and starch products: Part 2 Chemical methods (first revision).

^{*}Specification for A-twill jute bags (revised).

5.2 The material may also be packed in smaller containers lined with paper, or polyethylene which in turn shall have an outer protective packing.

6. MARKING

- **6.1** Each container shall be suitably marked so as to give the following information:
 - a) Name of the material;
 - b) Name and address of the manufacturer;
 - c) Batch or code number;
 - d) Net mass of contents; and
 - e) Any other information as required under Prevention of Food Adulteration Act, 1954.
- 6.1.1 Each container may also be marked with the ISI Certification Mark.

NOTE—The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks) Act and the Rules and Regulations made thereunder. The ISI Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by ISI and operated by the producer. ISI marked products are also continuously checked by ISI for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

7. SAMPLING

7.1 The method of drawing representative sample of the material and the criteria for conformity shall be as given in IS: 4662-1977*.

8. TESTS

- 8.1 The tests shall be carried out as prescribed in 4.2, 4.3 and in col 4, 5 and 6 of Table 1.
- 8.2 Quality of Reagents Unless specified otherwise, pure chemicals shall be employed in tests and distilled water (see IS:1070-1977†) shall be used where the use of water as a reagent is intended.

NOTE - 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

^{*}Methods for sampling of starches and starch products (first revision).

[†]Specification for water for general laboratory use (second revision).

APPENDIX A

[Table 1, Item (iii)]

DETERMINATION OF WATER-INSOLUBLE MATTER

A-1. APPARATUS

A-1.1 Preparation of Gooch Crucible — Digest a good, retentive grade asbestos with dilute hydrochloric acid (1:3). Wash free from acid and decant to remove fine particles. Prepare well-packed asbestos mat of suitable thickness in a gooch crucible. Wash with hot water, dry, ignite, rewash and dry at 135°C. Cool in a desiccator and weigh. Repeat washing, heating and drying to constant mass (alternatively, sintered glass filter, G-4 may be used).

A-2. PROCEDURE

A-2.1 Dissolve 10 g of the material in 200 ml of hot water and allow the solution to cool to room temperature. Filter through gooch crucible or G-4 sintered glass crucible (see A-1.1), wash with cold water until the washings are colourless and dry at $135\pm2^{\circ}$ C for three hours. Cool in a desiccator and weigh.

A-3. CALCULATION

A-3.1 Water-insoluble matter, percent by mass $=\frac{100 (M_2-M_1)}{M}$

where

 M_2 = mass, in g, of the crucible and the residue;

 $M_1 = \text{mass}$, in g, of the empty crucible; and

M = mass, in g, of the material taken for the test.

APPENDIX B

[Table 1, Item (v)]

DETERMINATION OF PHOSPHORUS CONTENT

B-1. PRINCIPLE

B-1.1 The organic substances are destroyed by digestion with sulphuric-nitric acid mixture and the phosphates are converted to orthophosphates. This forms into a phosphomolybdate, known as molybdenum blue, by means of a reducing agent. The intensity of the blue colour is measured photometrically at a wavelength of 825 nm.

B-2. REAGENTS

- **B-2.1 Sulphuric-Nitric-Acid Reagent** Prepared by mixing 1 part by volume of sulphuric acid (see IS: 226-1977*) having a relative density of 1.83 g/ml at 27°C corresponding to 96 percent by mass and 1 part by volume of nitric acid (see IS: 264-1976†) having a relative density of 1.38 g/ml at 27°C corresponding to 65 percent by mass.
- **B-2.2** Ascorbic Acid 50 g/l solution. This solution shall be kept in a refrigerator for a maximum period of 48 hours.
- **B-2.3 Molybdate Solution** In a 1-litre flask dissolve 10.6 g of ammonium molybdate tetrahydrate [(NH)₆ Mo₇O₂₄. 4H₂O] in 500 ml of water. Add 500 ml of sulphuric acid (10 N). Mix and leave to cool to ambient temperature.
- **B-2.4** Sodium Hydroxide Solution 10 N.

B-2.5 Phosphorus Solution

B-2.5.1 Stock Solution — Weigh to the nearest 0.5 mg, 0.439 5 g of anhydrous potassium dihydrogen phosphate and dissolve in water. Transfer quantitatively into a 1 000 ml volumetric flask. Dilute with water up to the mark and mix. This solution contains 100 μ g of phosphorus per millilitre.

Note — The potassium dihydrogen-phosphate shall be dried before use in a drying over at 105 $\pm 2^{\circ}C$ for one hour, and then cooled in a desiccator.

B-2.5.2 Standard Solution—Take 10 ml of the stock solution (see B-2.5.1) with a pipette and place in a 250 ml volumetric flask. Dilute to the mark with water and mix. This solution contains 4 mg of phosphorus per millilitre.

B-3. APPARATUS

- **B-3.1 Volumetric Flask** capacity 50, 100, 200 and 250 ml.
- B-3.2 Digestion Flask capacity 100 ml.
- **B-3.3 Pipettes** capacity 1, 2, 5, 10 and 15 ml.
- B-3.4 Conical Flasks capacity 50 ml.

^{*}Specification for sulphuric acid (second revision). †Specification for nitric acid (second revision).

B-3.5 Hot-Plate

B-3.6 Spectrophotometer or Colorimeter — with 1.0 cm cells, capable of measuring at a wavelength of 825 nm.

B-3.7 Water-Bath

- **B-3.8 Desiccator** with an effective desiccant.
- **B-3.9 Circulation-Type Cooling Bath** For temperature between 15 and 25°C.

B-3.10 Analytical Balance

B-4. PROCEDURE

B-4.1 Preparation of the Calibration Curve (From 0 to 40 µg of Phosphorus—Take a series of seven 50 ml conical flasks. Using a pipette, introduce into six of them, 1 0, 2 0, 3 0, 4 0, 5 0 and 10 0 ml of the standard solution corresponding to 4, 8, 12, 16, 20 and 40 µg of phosphorus. Add water to each of the seven flasks so that the total volume is approximately 30 ml and mix the contents. Using a pipette, add to each of the flasks, in the same order, 4 ml of the molybdate solution and 2 ml of the ascorbic acid solution. Mix after each addition. Place the seven flasks in a boiling water bath for 10 minutes. Then cool to ambient temperature by immersing the flasks in the cooling bath. Transfer quantitatively to the 50 ml volumetric flasks. Dilute with water to the mark and mix.

Using the spectrophotometer, determine the absorbance at 825 nm of each of the six solutions, using the solution from the flask without the standard solution as the reference. Plot the calibration curve giving the number of micrograms of phosphorus as a function of the absorbance.

- **B-4.2 Preparation of the Test Sample** Mix the sample thoroughly.
- **B-4.3 Test Portion** Weigh to the nearest 0.2 mg, 0.5 g of the test sample. This mass corresponds to an absorbance range between 0.1 and 0.7, if this is not the case, adjust the test portion accordingly (see Note).
- **B-4.4 Digestion** Transfer the test portion into a digestion flask. 7 Add 15 ml of the sulphuric-nitric acid reagent and mix well. Place the flask on the hot-plate. Heat gradually until the liquid is boiling gently in the flask. Continue boiling until the brown vapours are replaced by white vapours and the liquid has become clear. A persisting dark colour can be got rid

off by adding nitric acid drop by drop whilst continuing with the digestion. Leave to cool. Add 10 ml of water and get rid off the excess nitric acid by heating until the flask is filled with white vapours again.

- **B-4.5 Preparation of the Test Solution** Cool the mixture again and transfer the contents of the digestion flask to a volumetric flask with the help of 45 ml of distilled water. Raise the pH to 7.0 with the help of sodium hydroxide solution. Fill up to the mark with distilled water and mix thoroughly.
- **B-4.6 Determination** Take an aliquot (see Note) in a 50 ml-conical flask. Then using a pipette, add in the same order, 4 ml of the molybdate solution and 2 ml of the ascorbic acid solution, mixing after each addition. Place the flask in the boiling water bath for 10 minutes. Cool to ambient temperature by immersing the flask in the cooling bath. Transfer quantitatively to a 50 ml volumetric flask. Dilute with water up to the mark and mix. Determine the absorbance of this solution at 825 nm, by means of a spectrophotometer with a 1 cm cell and set at 825 nm. From the calibration curve read off the corresponding number of micrograms of phosphorus.
- **B-4.7** Blank Test Perform the blank test in duplicate, proceeding as given in B-4.4 to B-4.6, but omitting the test portion.
- **B-4.8 Number of Determinations** Perform two determinations on the same test sample.

B-5. CALCULATION

B-5.1 Total phosphorus content, percent by mass = $\frac{m_1 \times V_0 \times 100}{m_0 \times V_1 \times 10^6}$

where

 m_1 = phosphorus content in micrograms of the test solution as read from the calibration curve (B-4.1);

 m_0 = the mass, in grams, of the test portion (B-4.3);

 V_0 = dilution volume of the test solution (B-4.5); and

 $V_1 =$ aliquot volume used for the determination (B-4.6).

B-5.2 Take result as the arithmetic mean of the two determinations if the conditions of repeatability are satisfied,

B-5.3 Repeatability — The difference between the result of the two determinations carried out at the same time, by the same analyst on the same test sample (**B-4.2**) shall not exceed:

- a) 2 percent of their arithmetic mean in the case of phosphorus contents greater than 0.2 percent by mass, and
- b) 0.004 percent of phosphorus per 100 g of the product in cases where the phosphorus contents are less than 0.2 percent, by mass.

Note — For adjustment of the test portion to give the desired absorbance following dilutions may be made:

Phosphorus Content	Test Portion (B-4.3)	Dilution Volume (B-4.5)	Aliquot Volume (B-4.6)
(percent by mass)	g	ml	ml
Up to 0.05	0.500	100	2 5
More than 0.05 to 0.10	0.200	100	10
More than 0.10 to 0.25	0.200	100	2
More than 0.25 to 0.50	0.200	200	. 2

APPENDIX C

[Table 1, Item (vi)]

DETERMINATION OF VISCOSITY USING BROOKFIELD VISCOMETER

C-0. PRINCIPLE

C-0.1 The resistance to movement of a spindle is measured and expressed in terms of viscosity in seconds. The resistance being directly linked with viscosity can be expressed directly in terms of viscosity by previous calibration of the instrument.

C-1. APPARATUS

- C-1.1 Brookfield Viscometer Type LVF
- C-1.2 Mechanical Stirrer
- C-1.3 Constant Temperature Bath Maintained at 25°C.

C-2. PROCEDURE

C-2.1 Determine the moisture content of the sample. Calculate on dry basis, the mass of sample necessary to make 500 g test solutions as follows:

Mass of sample, in
$$g = \frac{100 \times A}{100 - B}$$

where

A = desired dry mass of sample in grams, and

B =percentage of moisture in the sample as weighed.

C-2.2 Add 100 ml distilled water to a jar, add the soluble starch phosphate and then add sufficient distilled water to make a total of 500 g of solution. Place the stirrer in the solution so that the blade is half way between the bottom of the jar and the surface of the liquid, stir till the sample dissolves. Remove the stirrer and transfer the sample container to the constant temperature bath and keep it there for 3 hours. Remove the sample container from the bath, stir it again and measure the viscosity with the Brookfield viscometer at 25°C selecting the proper spindle and the speed from Table 2. Allow the spindle to rotate until constant reading is obtained.

TABLE 2 VISCOMETER SPINDLES REQUIRED FOR GIVEN SPEEDS

Viscosity Range	Spindle No.	Speed rev/min	SCALE	FACTOR
(1)	(2)	(3)	(4)	(5)
10 to 100	1	60	100	1
100 to 200	1	30	100	2
200 to 1 000	2	30	100	10
1 000 to 4000	3	30	100	40
4 000 to 10 000	4	30	100	200

C-3. CALCULATION

C-3.1 Viscosity (Brookfield) in centipoises = Reading × Factor where

Reading = the number obtained from the viscometer, and

Factor = the number given in Table 2 for the spindle and speed selected.

C-3.2 Express the results in Pa.s (1 centipoise) = 10^{-3} Pa.s.

APPENDIX D

[Table 1, Item (viii)]

HEAVY METALS

D-0. GENERAL

D-0.1 The method for the determination of heavy metals is designed to determine those metallic impurities in the material that are coloured by hydrogen sulphide. The proportion of any such impurity is expressed as the quantity of lead required to produce a colour of equal depth in a standard solution for comparison, this quantity being stated as the heavy metals limit expressed as milligrams of lead per kilogram of the substance.

D-1. APPARATUS

D-1.1 Nessler Tubes — Two flat bottomed tubes of thin white glass, about 25 mm in diameter and about 150 mm in length, graduated at 50 ml. The depth, measured internally from the graduation mark to the bottom shall not vary by more than 2 mm in the tubes used.

D-2. REAGENTS

- **D-2.1** Delute Hydrochloric Acid 10 percent by volume.
- D-2.2 Bromine Solution Add to a small quantity of water 20 to 30 g of potassium bromide. Dissolve the potassium bromide in water and mix with it, 30 g of bromine Dilute the resulting mixture with water to produce 100 ml.
- D-2.3 Dilute Acetic Acid 6.0 percent by volume.
- **D-2.4** Stock Solution of Lead Dissolve 159'8 mg of lead nitrate in 100 ml of water to which one millilitre of concentrated nitric acid, specific gravity 1'42 (see IS: 264-1976*) has been added. Dilute this solution to 1000 ml with water. Store this solution in glass containers free from soluble lead salts.
- **D-2.5 Standard Lead Solution** Dilute 10 ml of the stock solution of lead, accurately measured, to 100 ml with water. This solution shall be freshly prepared, Each millilitre of this standard lead solution contains the equivalent of 0.01 mg of lead. When 0.1 ml of standard lead solution is employed to prepare the standard solution for comparison (**D-3.2**) with

^{*}Specification for nitric acid (second revision).

a solution of one gram of the material being tested, the solution for comparison thus prepared contains the equivalent of one part of lead per million parts of the material being tested.

D-2.6 Hydrogen Sulphide Solution — a freshly prepared saturated solution of hydrogen sulphide gas in water.

D-3. PROCEDURE

- D-3.1 Preparation of Solution of the Material Weigh accurately 2.5 g of the material into a porcelain dish and thoroughly char it over a Bunsen flame. Cool the dish, add to its contents 30 ml of dilute hydrochloric acid and 3 ml of bromine solution. Cover the dish with a watch-glass and boil the contents gently for 10 minutes. Filter the contents of the dish and wash the filter and residue with 25 ml of hot water. Collect the filtrate in a porcelain dish and evaporate to dryness. Dissolve the residue in 15 ml of water. Add 2 ml of dilute acetic acid, dilute to 25 ml with water and transfer to a Nessler tube.
- **D-3.2 Preparation of Standard Solution for Comparison** Introduce into another Nessler tube 2 ml of dilute acetic acid and 2.5 ml of standard lead solution and make up the volume to 25 ml with water.
- **D-3.3** To each of the two Nessler tubes, the one containing the solution of the material (**D-3.1**) and the other containing the standard solution for comparison (**D-3.2**) and 10 ml of hydrogen sulphide solution. Mix the contents of each Nessler tube separately, allow to stand for 10 minutes, then view the solutions downward over a white surface.

D-4. REPORT

D-4.1 The material shall be deemed to have satisfied the requirement if the colour produced by the solution of the material is not darker than that produced by the standard solution for comparison.